

The Dietary Supplement Ingredient Database (DSID) Botanical Initiative



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Introduction

The Nutrient Data Laboratory (NDL), Beltsville Human Nutrition Research Center (BHNRC), Agricultural Research Service (ARS) at USDA, in collaboration with the Office of Dietary Supplements, National Institutes of Health (ODS/NIH) and other federal agencies, has developed a Dietary Supplement Ingredient Database (DSID; <http://dsid.usda.nih.gov>) to evaluate levels of ingredients in dietary supplement products. The DSID is funded in large part by the Office of Dietary Supplements. It builds on the well-recognized strengths of the USDA/ARS in developing databases that support the assessment of intake of nutrients from foods. ODS provides leadership, jointly with its federal partners, in making this a reality. The consortium of federal agencies includes ODS and partners at USDA/ARS, the National Center for Health Statistics of the Centers for Disease Control and Prevention (NCHS/CDC), the Food and Drug Administration (FDA), the National Cancer Institute (NCI), NIH and the National Institute of Standards and Technology (NIST) of the Department of Commerce, who meet monthly as an advisory committee (Dietary Supplement Database Working Group).

Botanical Initiative



After completing studies on multivitamin/mineral (MVM) and omega-3 fatty acid dietary supplement products, a Botanical Initiative was launched in an effort to add botanical dietary supplements to the DSID. US National Health and Nutrition Examination Survey (NHANES) data show botanical dietary supplements as the 4th most common supplement type reported (see Table 1). The Dietary Supplement Database Working Group identified the 38 top non-vitamin/mineral bioactive ingredients in dietary supplements and considered these ingredients for analysis and inclusion in the DSID (Saldanha L et al., 2012). The ingredients were ranked based on these criteria: public exposure (intake and sales), the availability of validated analytical methods and analytical reference materials and scientific studies, economic and safety concerns. The top scoring 11 ingredients from this ranking process were: CoQ10, garlic, saw palmetto, ginkgo biloba, glucosamine, ginseng, green tea catechins (EGCG and other catechins), milk thistle, echinacea, flaxseed, and turmeric (curcumin).

Green tea and flavonoid-containing botanical supplements were chosen for initial study. Since these ingredients are also commonly found in foods, the data from these studies will complement the data for foods to track their combined consumption in US populations.

Pilot studies will evaluate methods of analysis by testing representative and top-selling products for prioritized ingredients of interest. Any quantitative issues for the analysis of extracts and mixed herbal blends and challenges associated with applying analytical results to various types of label information will be identified. Once the pilot studies are completed and a botanical sampling frame established, national studies of green tea and flavonoid-containing DS will be planned and implemented.

Table 1. US Prevalence of Use, Types of Dietary Supplements (Bailey et al., 2013)

Type of Supplement	Prevalence of use, NHANES (2007-2010), (%)
Multivitamin-mineral	31.9
Calcium	11.6
Omega-3/ fish oil	9.8
Botanical supplements	7.5
Vitamin C	7.1
Multivitamin	5.7
Vitamin D	4.9
Joint supplements	4.0
Vitamin E	3.7
Vitamin B12	3.3
Iron	1.8
Protein/sports	1.6
Folic acid	1.5
Fiber	1.1

Table 2. US Herbal and Botanical Consumer Sales by Channel (NBJ, 2013)

Herb and Botanical Product	2012 US Sales (\$ mil.)
1. Noni juice	245
2. Mangosteen juice	184
3. Green Tea	151
4. Saw Palmetto	134
5. Cranberry	126
6. Echinacea	124
7. Psyllium	118
8. Milk Thistle	115
9. Garlic	109
10. Turmeric	108
11. Multi-Herbs	107
12. Acai	105
13. Goji juice	101
14. Fruit & Vegetable Supplements	97
15. Ginkgo biloba	94

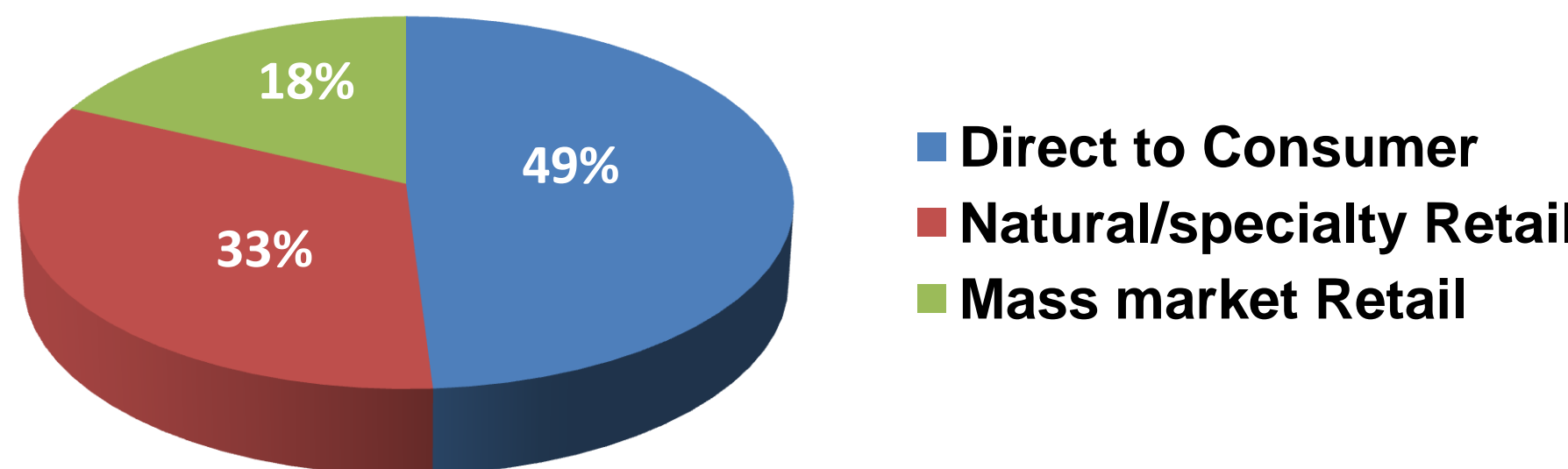


DSID Process for Botanical Studies

Sampling Plans

NDL plans to develop a national sampling frame for dietary supplements that can be applied to a variety of botanical supplements and modified for specific ingredients and product types. For botanicals, the market channel distribution is different from that seen with MVMs, where most products are purchased in mass market retail stores. NDL will identify and assess the information available about the US consumption and sales volume of these types of products. In addition to the current information used for sampling plans (NHANES, Nutrition Business Journal), strategies will be developed to identify and purchase representative samples of botanicals from direct and natural/specialty distribution channels. As the Dietary Supplement Label Database (DSLDD) continues to expand to incorporate all supplements, it will be essential for developing sampling plans, as it will provide a complete listing of products that can be sorted and categorized.

Figure 1. US Botanical Dietary Supplement Sales by Channel in 2012 (NBJ)



Analytical Methods

NDL will work with the Food Composition and Methods Development Laboratory (FCMDL), ARS, on research aspects of mutual interest. The FCMDL has determined the flavonoid content and variability in fruits, vegetables, and nuts sold in the U.S. (Harnly et al., 2006). In addition, scientists at FCMDL have completed the systematic quantitation of hydroxycinnamic acid derivatives and the glycosides of flavonols and flavones (Lin et al., 2012), and the systematic quantitation of flavanols, proanthocyanidins, isoflavones, flavanones, dihydrochalcones, stilbenes, and hydroxybenzoic acid derivatives based on UV molar relative response factors arising from the benzoyl structure (Lin and Harnly, 2012). Spectral and metabolomic fingerprinting of DSID samples will be investigated.

In addition, commercial laboratories will analyze botanical samples for ingredient content using methods validated with CRMs, spike recoveries, or other parameters. These methods will be optimized for the product types, ingredient ranges, and matrices identified as representative and/or top selling products.

Quality Control and Certified Reference Materials

NDL will add quality control (QC) materials to each batch of samples sent to labs, including product duplicates, in-house control materials, and certified reference materials (CRMs), if available. The results for these samples will be evaluated as indicators of laboratory precision and accuracy. Acceptable ranges for these materials will be established and reviewed for each batch in each study. The certified and reference values for NIST Standard Reference Materials (SRMs) green tea dietary supplements are listed in Table 3.

Table 3. Composition of NIST Green Tea SRMs

	SRM 3255	SRM 3256	SRM 3254
Ingredient	CV/RV (mg/g)	CV/RV (mg/g)	CV/RV (mg/g)
catechin	9.17	2.63	1.01
epicatechin	47.3	12	9
epicatechin gallate	100.3	17.1	12.7
epigallocatechin	81.8	30.7	25.2
epigallocatechin gallate (EGCG)	422	71.1	52
gallocatechin	22	7.55	2.4
gallocatechin gallate	39	4.6	99
epigallocatechin methyl gallate	6.87	--	--
gallic acid	3.231	13.1	1.12
caffeine	36.9	70	23.5
theobromine	0.867	1.04	0.463
theophylline	0.087	0.06	--
L-theanine	0.34	3.7	2.13

SRM 3255--Green tea extract
SRM 3256--Green tea solid oral dosage form
SRM 3254--Green tea leaf
CV/RV--Certified value/ reference value

Figure 2. Dietary Supplements with Green Tea reported in NHANES 2009-2010

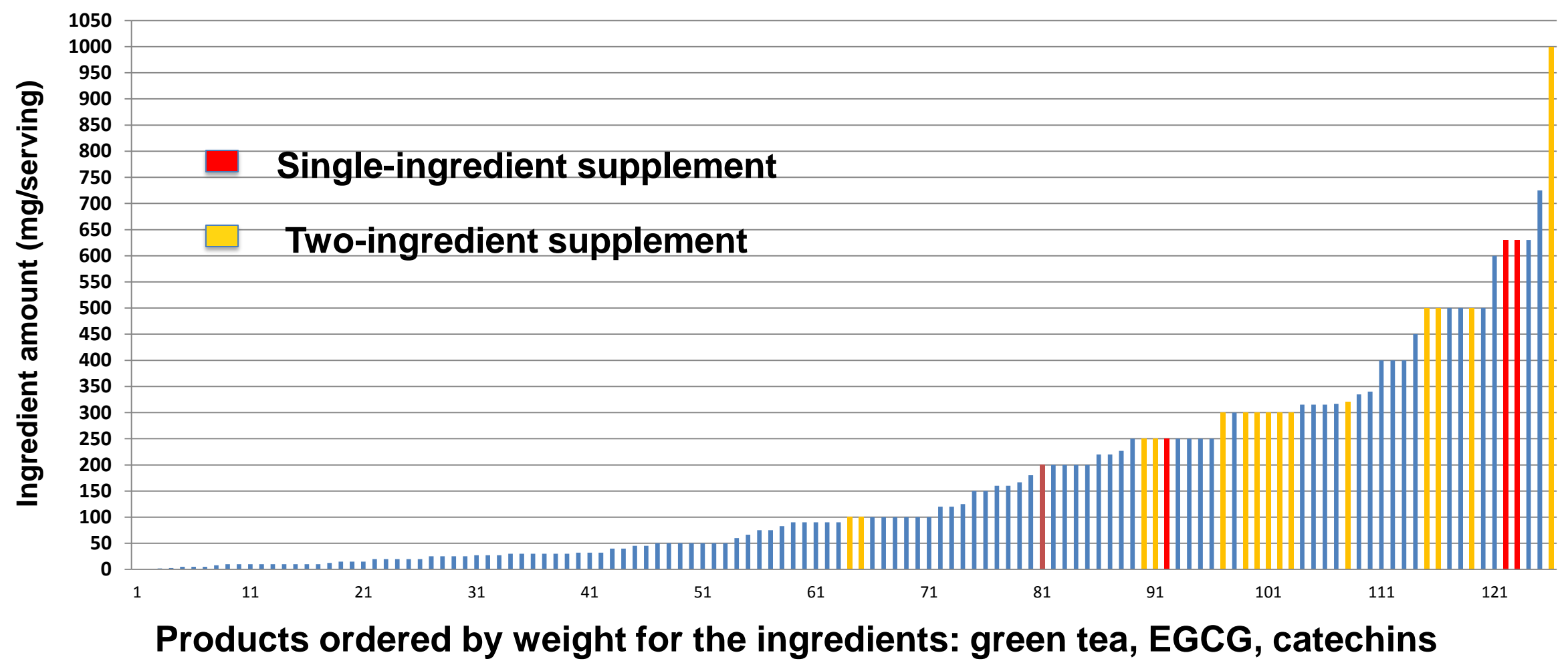
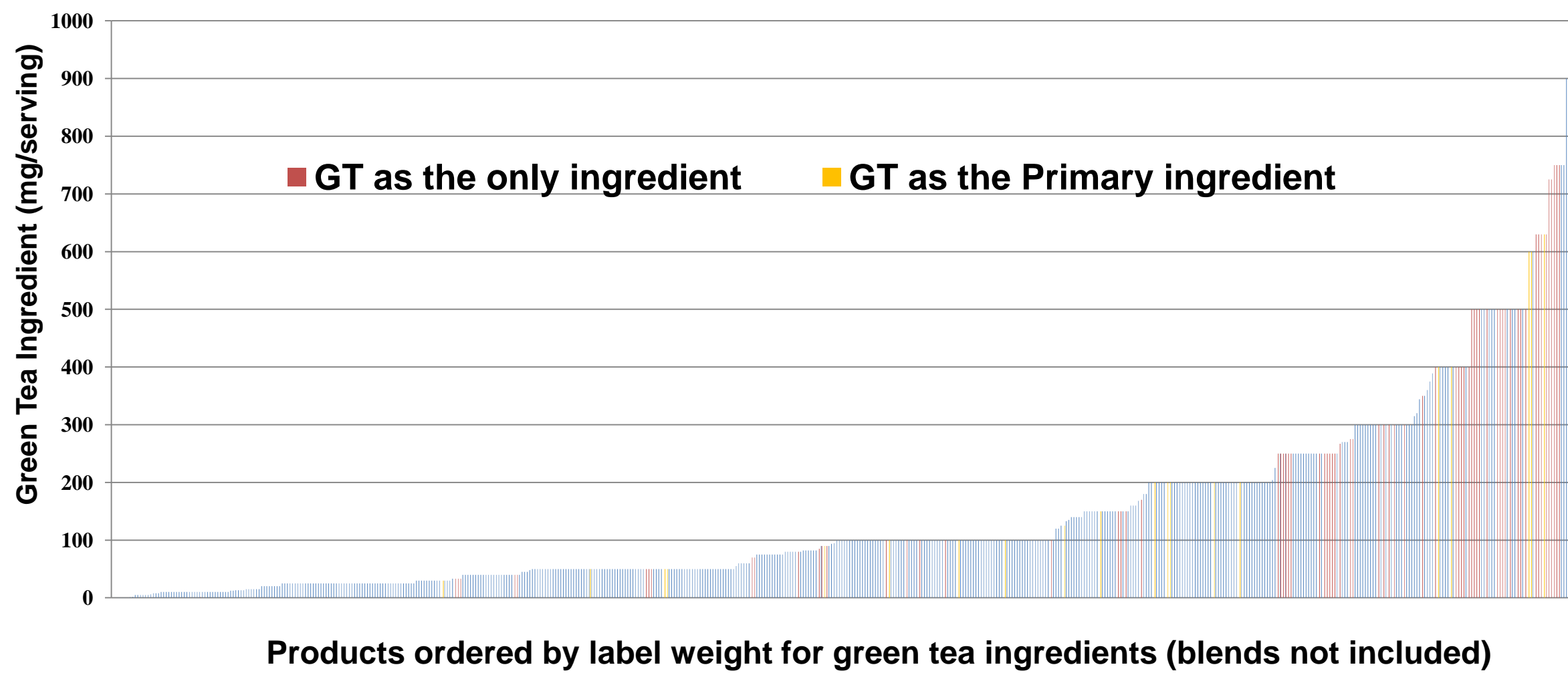


Figure 3. DSLD: Dietary Supplements with Green Tea (GT) (n=780)



Pilot Study: Catechins and Caffeine in Green Tea Supplements

Dietary supplements in NHANES and the DSLD were searched for products containing green tea ingredients (see Figures 2 and 3). After evaluating green tea containing products currently sold via varied channels including local stores (both mass market and natural/specialty markets), the internet and multi-level marketing, the decision was made to focus first on products with green tea as the only ingredient or the primary ingredient with no other botanicals in order to minimize interferences for analytical results and to compare to label claims for green tea components. The pilot study sampling plan was finalized and the goals were established:

1. Evaluate methods of analysis by testing representative and top-selling products for prioritized ingredients of interest
 2. Obtain estimates of content and variability for catechins, caffeine and other ingredients in green tea supplements
 3. Identify options for applying analytical results to various types of label information
 4. Determine the criteria and scope for the next study
- Three labs are participating in this pilot study. Currently, 2 lots of 33 different green tea products are being analyzed for the ingredients listed in Table 4. In addition, appropriate samples will also be tested for disintegration and for botanical identity.

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